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SCRAP RUBBER TIRE UTILIZATION IN ROAD DRESSINGS

Benson G. Brand

Battelle Columbus Laboratories

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Research to demonstrate the feasibility of using rubber from discarded passenger car tires in water-thinnable emulsions of asphalt or coal tar for blacktop dressings for driveways, etc., was conducted. Nine different compositions containing from 5 to 25 percent rubber were applied to field test site. After one year of continuous exposure in a high traffic area, the performance of the experimental compositions appeared to be as good as control samples. No attempts at composition optimization were made but complete feasibility of the basic idea has been successfully demonstrated.

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FOREWORD

Man and his environment must be protected from the adverse effects of pesticides, radiation, noise and other forms of pollution, and the unwise management of solid waste. Efforts to protect the environment require a focus that recognizes the interplay between the components of our physical environment--air, water, and land. The National Environmental Research Centers provide this multidisciplinary focus through programs engaged in

- studies on the effects of environmental contaminants on man and the biosphere, and
- a search for ways to prevent contamination and to recycle valuable resources.

The study described here was undertaken to demonstrate the feasibility of using rubber from discarded passenger car tires in emulsions as blacktop dressings for driveways. Employing rubber in this manner could increase the demand for used tires and decrease their solid waste disposal problem.

A. W. Breidenbach, Ph.D.
Director
National Environmental
Research Center, Cincinnati

EXECUTIVE SUMMARY

The objective of this study was to demonstrate the feasibility of using rubber obtained from discarded passenger car tires in water-thinnable emulsions of asphalt or coal tar for blacktop dressings for driveways, parking lots, streets, highways, etc. The use of rubber in this large volume market could result in an increased demand for the used tires, and, thus, decrease the solid waste disposal problems connected with 200 million discarded tires per year.

The study has resulted in the production of nine different compositions containing from 5 to 25 percent rubber that were promising enough to apply to a high-traffic area in the BCL parking lot (estimated passage of 1800 cars per day), to determine the respective service lives of the compositions.

After exposure for one year under the above conditions, performance appears to have been as good as that of the control samples, and protection of the underlying blacktop is still being rendered by all samples.

The goal of the project was the demonstration of the feasibility of the basic idea that rubber salvaged from discarded auto tires could be incorporated into water-thinned blacktop dressings, and to utilize the information obtained to generate interests of industrial companies continuing the studies to result in marketable products.

It is felt that the goals of this study have been fully met. Complete feasibility of the basic idea has been demonstrated. Service-life demonstration have shown good performance characteristics, even though no attempts were made to optimize the formulation. One proposed research program was supplied to a government agency at their request. This was technically approved but funding could not be arranged. Two additional proposals have been submitted to industrial companies, and are currently under consideration.

Future plans for Battelle activity in this area will involve attempts to interest industry in continuing the study to develop improved materials.

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INTRODUCTION

Battelle's Columbus Laboratories has carried out research under Grant No. R-EP-00500-01 having the following objective: to explore the feasibility of effective and advantageous use of ground scrap or reclaimed rubber from discarded rubber tires that now present a sizeable disposal problem. The research was directed toward this objective by studying the development of improved water-thinned blacktop dressings through incorporation of the rubber salvaged from used passenger car tires in a bituminous (asphalt or coal tar) base.

SUMMARY

An exploration of the rubber industry has shown a wide variety of products is available or can easily be made available from discarded automobile tires that might have potential in producing materials for use in blacktop dressing manufacture. Our laboratory studies under this grant have shown that it is possible to blend finely ground tire rubber into asphalt or coal tar by either of two

processes. The resulting blends have been satisfactorily emulsified to form materials suitable for use as blacktop dressings. Nine different blends containing from 5 to 25 percent rubber have been made into emulsions and applied to a blacktop surface in a high-traffic area (1800 cars/day).

Evaluation of the outdoor exposures after one year shows good performance for all samples.

Accelerated exposures in the Weather-Ometer indicate differences exist, with some samples showing better performance than the controls containing no rubber.

Slip-resistance test indicate a slight superiority for the rubber-containing samples, both wet and dry.

Formulas for the nine experimental products appear in the Appendix.

BACKGROUND

Approximately 250 million new automobile tires are manufactured every year. The ultimate destination of a large portion of these tires is the trash heap, where they pose a major disposal problem. Disposal by burning contributes to air pollution. Tires produce unstable landfill, and, hence, are frequently banned from this method of disposal.

A sizeable number of tires are retreaded and reused. The percentage of truck and bus tires that are retreaded is considerably higher than that for passenger car tires. However, the number of retreaded tires is of little consequence, since the retreading process only delays the discard date, and the retreaded tire still ends up as a solid waste disposal problem.

A considerable tonnage of rubber polymer is "reclaimed". The reclaiming process involves the separation of fabric and steel wire bead from the rubber polymer, followed by digestion or depolymerization, and blending with reclaiming oils. This results in a product that can be used for recycling back into tire manufacture, tire retreading, and many noncritical materials, such as elastic webbing, floor mats, sink mats, rubber hose, etc.

Present use of tires for the reclaiming industry accounts for about 20 percent of the tires produced (about 50 million tires per year). Major inputs to this industry are rubber factory scrap, tire buffings, and inner tubes. Economic trends indicate a decreasing market for reclaimed rubber, because several grades of virgin rubber are now selling for less than reclaimed rubber. This is especially true if one considers that reclaimed rubber is usually only about 50 percent rubber polymer, the remainder being reclaiming oils.

Considerable interest over the years has been centered on "rubberized roads". The technical literature is full of papers describing the use of rubber with asphalt in the construction of highways. These papers are presently limited to two modes of usage. The most prevalent use is the blending of chopped-up tires with asphalt for use as a paving composition. The chopped tires replace a large portion, or all of the aggregate (crushed rock or gravel) in the paving composition. Several state highway departments are known to be exploring this application. Advantages claimed for rubberized roads prepared by this technique include decreased skidding, longer road life, and decreased cracking and spalling of the surface.

The second mode of rubberizing roads described in the literature involves the addition of rubber latex (unvulcanized) to the hot-mix asphalt paving composition. The same advantages are claimed as for the above procedure. However, it must be pointed out that this procedure is more costly than using reclaimed rubber, and the unvulcanized rubber tends to contribute less to the durability than does vulcanized rubber.

Several valuable bibliographies on the use of rubber in roads have been prepared. One particularly valuable list of references, which is kept up to date, is available from

Highway Research Board Library
National Academy of Sciences
2101 Constitution Avenue
Washington, D.C. 20418
Attention D. Bright, Librarian.

A review of these available bibliographies plus a literature search by Battelle personnel prior to this study has failed to disclose a single reference to the emulsification of a dispersion of vulcanized rubber in either asphalt or coal tar, for use as a blacktop dressing for coating driveways, parking lots,

etc. The closest approach found was the use of an emulsified rubber-asphalt system as a bond coat between concrete and hot-mix asphalt road surfacing, to decrease "reflective cracking".

INDUSTRY SURVEY

The study covered by this report was initiated by a survey of the rubber reclaiming industry to identify materials that could be incorporated into this program. Valuable telephone and mail communications with Midwest Rubber Reclaiming Company, E. St. Louis, Illinois; U. S. Rubber Reclaiming Company, Vicksburg, Mississippi; and Uniroyal, Naugatuck, Connecticut, were especially helpful in the early phases. The Asphalt Institute, College Park, Maryland, furnished excellent background information.

The Rubber Reclaimers Association, Naugatuck, Connecticut, was most helpful. Through the cooperation of Mr. Tom Fitzgerald, Executive Secretary, it was possible to arrange for the Association to supply six samples of reclaimed rubber for use in this study. These samples were selected to be representative of the entire industry. They were prepared by Mr. C. E. Stuecheli of Xylos Rubber Company, Akron, Ohio. (This company is no longer in existence.) Mr. Louis Baker, President of A. Baker Manufacturing Company, has also been most helpful in supplying samples of low-cost ground tires, as well as depolymerized rubber.

Mr. C. A. Hauck of Union Carbide Corporation, Tarrytown, New York, has been helpful in supplying freeze-ground tire samples.

Mr. Charles McDonald of the City of Phoenix, Arizona, has graciously supplied samples of his high-temperature dispersion of rubber in asphalt for our emulsification studies, as well as excellent instructions for carrying out his process.

Dr. E. E. McSweeney of Union Camp Corporation, Savannah, Georgia, supplied samples on high and low penetration tall oil pitch which were also used in this study.

DESCRIPTION OF SAMPLES

Rubber Reclaimers Association Samples

As mentioned above, through the courtesy of Mr. Tom Fitzgerald, Executive Secretary of the Rubber Reclaimers Association, arrangements were made to obtain six samples of reclaimed rubber, representative of materials that could be made by any reclaimer. Three compositions were to be made available in both "slab" and "crumb" form. These three compositions (six samples) were prepared by Mr. C. E. Stuecheli, of Xylos Rubber Company, Akron, Ohio, 44301. (This company is no longer doing business.)

Properties of the six samples, as furnished by Mr. Stuecheli, (slab and crumb form) are recorded as follows:

	Sample No.		
	1x (Slab & Crumb)	2x (Slab & Crumb)	3x (Slab & Crumb)
Scrap (35 mesh) Defiberized	100	100	100
Oil Crowley[a]	8	8	12
Chem. Softener Pitt-Consol 500	1	1	1
Talc	5	5	5
Microcel E (in crumb only)	2	2	2
Cooking Time @ 375 F (hrs)	6	10	6
Sp.G.	1.175	1.175	1.170
Mooney ML/4 (on refined scrap)	58	65	60
Ash %	8.9	8.9	8.8
Acetone Extract	21.5	21.0	24.8
Chloroform Extract	7.8	7.2	7.6

	Sample No.		
	1x (Slab & Crumb)	2x (Slab & Crumb)	3x (Slab & Crumb)
Carbon Black	26.1	27.3	26.4
Approx. RHC(b)	40	40	37

(a) Rubber reclaiming oil.
(b) Rubber hydrocarbon content.

No specific price quotations were given for the above samples. The general understanding was that the price would be in the neighborhood of 9 to 14 cents per pound, depending on the volume demand, the cooking (processing) time, and whether the product was desired in the "crumb" or "slab" form. Processing of the samples into the "slab" form represents an additional step in the manufacturing, and, hence, increases the cost per pound.

Union Carbide Sample

Mr. C. A. Hauck of the Linde Division, Union Carbide Corporation, Tarrytown, New York, 10591, supplied a sample of cryogenically ground whole passenger tires. This sample had the following particle size distribution:

U.S. Mesh Size	Nominal wt, percent past
-100	100%
-200	50%
-325	5%

It was prepared from whole passenger car tires (tread plus sidewalls). It contained no oils or other reclaiming agents. It was estimated that at least 90% of the fiber had been removed by the preparation process used. The projected price was calculated to be 4 to 10 cents per pound, probably nearer the 4-cent range. Union Carbide's code for this sample is UCC-B-WT-1.

Baker Samples

The A. Baker Manufacturing Company, South Bend, Indiana, supplied three samples of ground passenger car tires for our studies. One of these

samples was considered to be unsatisfactory for our use, based on its particle size. The particles in this sample were roughly 1/2 inch x 1/2 inch. The two remaining samples proved to be worthy of investigation. One sample was ground whole passenger car tires with an estimated 75% of the fabric removed after grinding. The particle size was similar to that of the Union Carbide sample. A sieve analysis furnished by the supplier indicated it to be -12, +16 mesh. The price of this material is expected to be in the 4 to 5 cents per pound area, which might be reduced for large volume uses.

The third sample was labelled as "depolymerized" rubber. This sample had been cooked and blended with reclaiming oils and surfactants. The exact composition is considered to be proprietary. However, the reclaiming oils are suspected to contain significant amounts of terpenes, disulfides, and highly polar swelling agents added to improve the handling properties of the rubber.

The particle-size range of the sample appeared to be about the same as that of the finely ground sample, approximately -12, +16 mesh. The presence of fabric was not nearly as apparent as it was in the ground rubber.

The depolymerization requires extra processing. The price is, therefore, in the 7 to 8 cent per pound range. If we assume this sample to be 75% rubber polymer and 25% reclaiming oils, the price is about 10 cents per pound of rubber polymer, which is considered to be high for the intended application.

Phoenix Sample

Mr. C. H. McDonald, Engineering Supervisor, City of Phoenix, Arizona, 85034, supplied a sample of rubber that had been incorporated into asphalt by a heating process developed by him for use in patching and pavement repair compounds. This sample was a 25% dispersion of ground tire rubber (-16, +25 mesh) in 120-150 penetration grade Los Angeles Basin asphalt.

The incorporation process involves heating the mix until a reaction occurs, yielding a jelly-like composition. A patent application has been filed by McDonald covering the process.

Mr. McDonald has not tried to emulsify the material. Its use has been restricted to paving and pavement patching using the hot-mix technique, and a hot-melt spray coating for streets.

Care must be used not to overheat the sample, since depolymerization of the rubber takes place at about 400 F, which injures the durability of the product, according to Mr. McDonald.

Uniroyal Dispersion

Uniroyal Chemical, Naugatuck, Connecticut, 06770, markets a water dispersion of reclaimed rubber. This dispersion is 58% solids, and is anionic in nature. The solids content of the dispersion was

Component	Parts
Ground Whole Passenger Tires	100
Tall Oil Pitch	4
Bituminous Petroleum	3
Asphaltic Resin	5
Oleic Acid	9

The price, in large quantity, can be expected to be about 20 cents per dry pound.

The sample appeared to be of medium viscosity. Suspended particles appeared large, much larger than in an emulsion.

Although this sample was anionic in nature, it was found to be incompatible with separately prepared emulsions of either asphalt or coal tar made by normal procedures.

Based on the high price and the difficulty of incorporating it in blends with asphalt or coal tar, this sample was judged to be unsuitable for this study.

Porter Sample

The sample received from H. K. Porter Company was chopped rubber hose, in the form of rings. It contained considerable fabric. The particles were too

large to disperse readily into either asphalt or coal tar. The sample was, therefore, judged not suitable for use in this study.

Tall Oil Pitch

The Union Camp Corporation, Savannah, Georgia, 31402, furnished two representative samples of tall oil pitch, high and low penetration. The analyses of these samples were as follows

	High Penetration	Low Penetration
Acid Number	64	31
Saponification Number	109	97
Rosin Acids %	28.2	15.1
Fatty Acids %	39.7	48.1
Unsaponifiables	32.1	36.8

The presence of rosin acids, fatty acids and terpenes in tall oil pitch make it appear interesting as a dispersing agent for vulcanized rubber.

Asphalt Emulsion Base

The asphalt used as the base in this study is a petroleum residue asphalt with a penetration of 190. It was obtained from Ashland Chemical Company as representing their recommendation for water emulsion use.

Coal Tar

The coal-tar base used in this study was obtained from Reilly Tar & Chemical Corporation. It is a standard emulsion-type base, sold by the supplier to many blacktop-dressing manufacturers.

EXPERIMENTAL

Incorporation of Rubber into Asphalt and Coal Tar

Several methods were investigated to incorporate the various types of rubber received into asphalt and coal tar. The first and one of the most promising methods was to place the asphalt or coal tar into a jacketed Bramley mill and add the rubber during agitation. The best technique was to heat the mill, after addition of the asphalt or coal tar, to 265 to 275 F with 45 to 50 psig pound steam. When the asphalt or coal tar was liquid, the rubber was added. After sixty minutes, the steam was turned off and cold water at 40 to 45 F was circulated through the jacket for an additional sixty minutes. This cycle was repeated four times. The Bramley mill is a modified sigma-blade mixer which places high shear and tearing action on thick mastic materials. This action which occurred during the cold-water segment of the cycle tore apart the rubber particles which had been swollen due to the heat of the first half of the cycle and the solvency of the oils in the asphalt and/or coal tar.

The second method tried for incorporating rubber into asphalt and coal tar was to use a 6-inch rubber mill. This method resulted in a higher percentage of rubber being incorporated into the asphalt and the coal tar, but it was a much more expensive process than any of the other methods explored. The rubber was milled on heated rolls until it was in sheet form. Then a small amount of asphalt or coal tar was added. The greatest reduction of the rubber that could be obtained was a composition containing 25% asphalt and 75% rubber. This mixture was very difficult to remove from the mill even when the rolls were cooled to 50 F. Further reduction of the rubber content was made by heating the rubber-asphalt mixture until liquid, then adding more liquid asphalt until a 5% rubber-asphalt mixture was obtained. There did not appear to be any major differences in properties of the rubber-asphalt and rubber-coal tar blends made by this method and the first method using the Bramley mill; therefore, due to economics, this method was not investigated further.

The third method investigated was to swell the rubber in tall oil before the addition of asphalt or coal tar. This work was done in the Bramley

mill using the same cycles as described in the first method. By swelling the rubber in the tall oil before adding the asphalt or coal tar, the final mixtures had smoother texture and the rubber appeared to be better incorporated into the asphalt and coal tar; however, the high oil content made these mixtures very difficult to emulsify.

The fourth method used to incorporate the rubber was the Phoenix method. This process method was developed by Mr. Charles H. McDonald, Engineering Supervisor, Engineering Department, Materials Testing Station, City of Phoenix, Arizona, 85034. This process was to heat the asphalt to 350 F and add 25 % ground rubber (-16 mesh, +25 mesh) to the heated asphalt during agitation. Mr. McDonald emphasized that the mixture should not be heated over 400 F or the rubber would depolymerize. Mr. McDonald stated that the rubber reacted with the asphalt to form a jelly-like composition. A patent has been applied for by Mr. McDonald. Our experiments showed that the 350 F temperature was fine for incorporation of rubber into asphalt but slightly higher temperatures (375 to 380 F) were needed to get the rubber into coal tar.

It was decided, based on economics, ease of incorporation, and appearance of the mixtures of rubber-asphalt and rubber-coal tar, to use the methods designated one and four. The following table lists the name of the rubber used, percentages of rubber added, method of incorporation, and the base material (asphalt or coal tar) to which the rubber was added.

TABLE 1. INCORPORATION OF RUBBER INTO ASPHALT AND COAL TAR

Rubber Sample (Name)	Rubber Type	% Added	Method (a)	Base Material
Baker	Fine ground tires w/fibers	5 + 25	1 + 4	Asphalt & Coal tar
Baker	Full reclaim	5 + 25	1 + 4	Asphalt & Coal tar
Xylos 1x	Full reclaim	5 + 25	1 + 4	Asphalt & Coal tar
Xylos 2x	Full reclaim	5 + 25	1 + 4	Asphalt & Coal tar

TABLE 1. (Continued)

Rubber Sample (Name)	Rubber Type	% Added	Method[a]	Base Material
Xylos 3x	Full reclaim	5 + 25	1 + 4	Asphalt & Coal tar
Union Carbide	Cryogenic Ground 90% Fibers removed	5 + 25	1 + 4	Asphalt & Coal tar

(a) 1 - Battelle method (Bramley mill)
4 - Phoenix method.

The Baker fine ground rubber with fiber was incorporated into asphalt and coal tar with no difficulties by both the Battelle and Phoenix methods at both the 5 and 25 percent level.

The Baker full reclaim rubber was extremely difficult to incorporate by the Phoenix method when added to either the hot asphalt or hot coal tar at both the 5 and 25 percent level; the rubber would cause heavy foaming and in most cases foamed out over the container. This reaction was most likely caused by boiling of the oils used to reclaim the rubber.

Also the Baker reclaimed rubber was fairly difficult to incorporate into asphalt and coal tar at the 5 percent level in the Bramley mill and the resulting mixture was granular in appearance. At the 25 percent level the Baker reclaimed rubber had lumps about 1/4 inch in diameter.

Xylos 1x crumb was the only one of the three reclaimed rubbers from Xylos Rubber Company that could be incorporated smoothly into the asphalt and coal tar in the Bramley at both the 5 and 25 percent level. The other two rubbers, Xylos 2x and Xylos 3x, looked similar to the 5 percent Baker reclaim. They all had a very granular texture. All the Xylos crumb rubbers were incorporated into asphalt and coal tar by the Phoenix method and looked good at both levels of addition (5 and 25 percent).

The Union Carbide cryogenic ground rubber was easy to incorporate into both asphalt and coal tar by both methods at the 5 and 25 percent levels.

All three slab rubber samples proved to be very difficult to disperse in both asphalt and coal tar, and their use was abandoned.

Emulsification of Rubberized Asphalt

All asphalt emulsification work was conducted with an Asphalt Emulsion Base obtained from Ashland Chemical Company. During the first attempts to emulsify the asphalt, various clays and talcs were used as fillers and Indulin W-1 was used as an emulsifying agent. Most of these emulsions were very poor and heavy settling occurred when the emulsions stood overnight. The most stable emulsions resulted when Mistron ZCS talc was used as the filler. The following was selected as the standard method for evaluating rubberized asphalt.

Three hundred grams of water and 100 grams of United Sierra's Mistron ZCS talc were placed in a quart container. A Premier mill mixer was used to provide high-speed agitation (∿5000 rpm) of the water-talc slurry. The slurry was heated to 160 to 170 F during constant agitation. Two grams of calcium chloride and 85 to 90 grams of hot (180 to 200 F) asphalt-rubber mixture were added. As soon as the addition of asphalt-rubber mixture was made, the agitation was increased to 7500 to 8000 rpm for one minute. Table 2 lists the asphalt-rubber mixtures emulsified and comments on their stability and appearance.

TABLE 2. EMULSIFIED ASPHALT-RUBBER MIXTURES

Asphalt-Rubber	% Rubber	Method[a]	Comments	
			Appearance	Stability
Xylos 1x	5	BCL	Very Good, Smooth	Good
Xylos 1x	25	Phoenix	Good, Smooth	Good
Xylos 1x	5	Phoenix	Good, Smooth	Good
Xylos 1x	25	BCL	Good, Slightly Granular	Good
Union Carbide	5	Phoenix	Excellent, Very Smooth	Very Good
Union Carbide	5	BCL	Excellent, Smooth	Very Good
Union Carbide	25	Phoenix	Good, Smooth	Good
Union Carbide	25	BCL	Good, Smooth	Good
Baker Reclaim	5	BCL	Good, Slightly Granular	Fair

TABLE 2. (Continued)

Asphalt-Rubber	% Rubber	Method[a]	Comments	
			Appearance	Stability
Baker Reclaim	5	Phoenix	Good, Slightly Granular	Good
Baker Reclaim	25	Phoenix	Fair, Lumpy	Fair
Baker Reclaim	25	BCL	Fair, Granular	Good
Baker Ground	5	BCL	Very good, Very Smooth	Good
Baker Ground	25	Phoenix	Good, Smooth	Good
Baker Ground	5	Phoenix	Good, Smooth	Good
Baker Ground	25	BCL	Good, Very Slightly Granular	Good
Phoenix-Asphalt Rubber Mix	25	Phoenix	Fair, Lumpy	Fair
Xylos 2x	5	Phoenix	Good, Slightly Granular	Fair
Xylos 2x	5	BCL	Fair, Granular	Fair
Xylos 3x	5	Phoenix	Fair, Granular	Fair
Xylos 3x	5	BCL	Fair, Granular	Fair

(a) BCL - Battelle's Columbus Laboratories.

Rubberized Coal Tar Emulsification

All coal tar emulsification work was conducted with a coal tar emulsion base from Reilly Tar and Chemical Company. Initial work on emulsification was to evaluate several different clays and emulsifiers. The following list gives the clays and emulsifiers tried.

Clays	Supplier
Hydrite PXS	Georgia Kaolin Co.
Kaogen 45	" " "
Hydrite RS	" " "
Kaophobe 45	" " "
#20 Clay	Burgess Pigment Co.
Hydrite Flat "D"	Georgia Kaolin Co.

Emulsifiers	Supplier
Kricinol 35	Arthur C. Trash Co.
Indulin C	Westvaco Corp. Chem. Div.
Indulin W-1	" " " "
Westvaco M-50	" " " "
Westvaco Resin 90	" " " "
Arizona DRS 42	Arizona Chemical Co.
Arizona DRS 43	" " "
Dresinate X	Hercules, Inc.
Dresinate XX	" "
Redicote E-1	Armour Industrial Chemicals
Redicote E-3	" " "
Redicote E-5	" " "
Redicote E-9	" " "
Redicote E-11	" " "
Redicote E-12	" " "
Redicote E-14	" " "
Redicote E-23	" " "
Redicote E-27	" " "
Redicote E-28	" " "

The following was selected as the standard method for the evaluation of rubberized coal tar. One hundred and twenty grams of water, 20 grams of Burgess #20 #20 clay, and 10 grams of Mistron ZCS Talc were placed in a quart container. A Premier mill mixer was used to provide high-speed agitation (~5000 rpm) of the water-clay-talc slurry. The slurry was heated to 160-180 F with constant agitation. When the slurry reached the desired temperature, 1 gram of Indulin W-1 and 1 gram of calcium chloride was added slowly to the slurry during constant agitation.

When approximately 50 percent of the rubberized coal tar had been added the mixer speed was increased to 7500-8000 rpm. After all the rubberized coal tar was added, 1 gram of potassium ricinoleate was added. Before the addition of the potassium ricinoleate the rubberized coal tar was suspended in little globules in the slurry. The addition of the potassium ricinoleate caused the breaking up of the globules and resulted in a good emulsion. Table 3 lists the coal-tar rubber mixtures emulsified and comments on their stability and appearance.

TABLE 3. EMULSIFIED COAL-TAR RUBBER MIXTURES

Coal-Tar Rubber	% Rubber	Method	Comments	
			Appearance	Stability
Xylos 1x	5	BCL	Good, smooth	good
Xylos 1x	25	Phoenix	Good, slightly granular	good
Xylos 1x	5	Phoenix	Good, very slightly granular	good
Xylos 1x	25	BCL	Good, slightly granular	good
Union Carbide	25	Phoenix	Good, smooth	good
Union Carbide	5	Phoenix	Good, smooth	good
Union Carbide	5	BCL	Good, smooth	good
Baker Reclaim	5	Phoenix	Fair, granular	fair
Baker Reclaim	5	BCL	Good, granular	good

TABLE 3. (Continued)

Coal-Tar Rubber	% Rubber	Method	Comments	
			Appearance	Stability
Baker Ground	25	Phoenix	Good, very slightly granular	good
Baker Ground	5	Phoenix	Good, smooth	good
Baker Ground	5	BCL	good, smooth	good
Baker Ground	25	BCL	good, very slightly granular	good
Xylos 2x	5	Phoenix	Fair, granular	fair
Xylos 2x	5	BCL	Fair, granular	fair
Xylos 3x	5	BCL	Poor	poor
Xylos 3x	5	Phoenix	Fair, granular	fair

Preparation of Experimental Emulsions for Exterior Evaluations

Asphalt

Five experimental asphalt-rubber emulsions and four experimental coal-tar rubber emulsions were prepared in 4-gallon lots for exterior exposure evaluation. Table 4 designates the experimental emulsions made.

TABLE 4. EXPERIMENTAL EMULSIONS FOR EXTERIOR EXPOSURE

Emulsion Number	Rubber	% Rubber	Method[a] Rubber Incorporation	Base Material
TE-1	Xylos 1x	5	BCL	Asphalt
TE-2	Baker Fine	5	BCL	Asphalt

TABLE 4. (Continued)

Emulsion Number	Rubber	% Rubber	Method[a] Rubber Incorporation	Base Material
TE-3	Union Carbide	5	BCL	Asphalt
TE-4	Baker Fine	25	Phoenix	Asphalt
TE-5	Baker Fine	5	Phoenix	Asphalt
TE-6	Xylos 1x	5	BCL	Coal tar
TE-7	Union Carbide	5	BCL	Coal tar
TE-8	Union Carbide	25	Phoenix	Coal tar
TE-9	Baker Fine	5	BCL	Coal tar

(a) BCL - Battelle's Columbus Laboratories.

All the emulsions were made on a laboratory model Cowles Dissolver. The asphalt control and five experimental emulsions were formulated as follows.

Added to Dissolver tank

9 kilograms water

3 kilograms Mistron ZSC talc

60 grams Calcium Chloride

Heated to 160 F with constant agitation then added 4.5 kilograms asphalt or asphalt-rubber mix heated to 200 to 210 F during constant agitation.

Coal Tar

The four experimental coal-tar rubber emulsions were formulated as follows.

Added to Dissolver tank

7.2 kilograms water

1.2 kilograms Burgess #20 Clay

.6 kilograms Mistron ZSC talc

60 grams Indulin W-1

60 grams Calcium Chloride

Heated above to 180 F with constant agitation then added 5.4 kilograms coal tar-rubber mix.

Heated to 220 to 230 F during constant agitation. Immediately after all the coal tar-rubber mixture was added, 60 grams of potassium ricinoleate was added.

All the experimental emulsions were smooth and looked good. After standing for 14 days, TE-4 had a granular appearance and it was fairly difficult to reincorporate the surface liquid. TE-8 was extremely difficult to stir; however, after the surface liquid was reincorporated, it had a very smooth texture. All of the other experimental emulsions mixed as well as the coal tar and asphalt controls.

Weather-O-Meter Studies

Table 5 lists the emulsions that were applied to 2-1/2-inch x 6-inch asbestos boards for evaluation in the Weather-O-Meter. The twin-arc Weather-O-Meter was cycled so that the panels would have 108 minutes dry, high temperature (100 to 120 F) and 12 minutes of water spray directly on the panels.

Evaluation of the Weather-O-Meter exposed samples is given in Table 5.

Weather-O-Meter Evaluations

It was noted early in the exposure life of the panels in the Weather-O-Meter, that atypical behavior was being encountered. The changes observed did not agree with the noted performance of the samples applied to Battelle's driveway. Failure by forming large alligator-skin-like cracks was almost universal.

This variance in performance is felt to be due to the facts that

(1) The samples in the Weather-O-meter series are much thicker (1/8 inch) than the driveway samples.

TABLE 5. WEATHER-O-METER EVALUATION OF EXPERIMENTAL EMULSIONS

Specimen Number	Emulsion Number	Comp Components	% Rubber	Method of Rubber Incorporation(a)	100 hr Evaluation Comments	1000 hr Evaluation Comments
1, 2	TE-1	Asphalt-Xylos 1x	5	BCL	Very slightly checked smooth film	Smooth, very slightly checked gray
3, 4	TE-2	Asphalt-Baker fine	5	BCL	Slightly checked soft film, few rubber chunks	Slightly checked whitish, few rubber chunks
5, 6	TE-3	Asphalt-Union Carbide	5	BCL	Medium checking mud cracking, dull film, soft, rubbery	Considerable checking, whitish eroded
7, 8	TE-4	Asphalt-Baker fine	25	Phoenix	Very rough rubbery, heavy cracks	Rough, slight cracks, rubbery
9, 10	TE-5	Asphalt-Baker fine	5	Phoenix	Smooth film, gray rubber chunks	Smooth, gray, very slighly checked
11, 12	TE-6	Coal tar-Xylos 1x	5	BCL	Thin, heavy alligatoring medium-hard film	Dull, heavily alligatored, pinholed
13, 14	TE-7	Coal tar-Union Carbide	5	BCL	Very heavy alligatoring, flat film, medium hard film	Dull, very heavily alligatored, pinholed
15, 16	TE-8	Coal tar-Union Carbide	25	Phoenix	Gray, hard, no cracks, few craters	Slight surface cracks, whitish pinholed

TABLE 5. (Continued)

Specimen Number	Emulsion Number	Components Components	% Rubber % Rubber	Method of Rubber Incorporation(a)	100 hr Evaluation comments	1000 hr Evaluation Comments
17, 18	TE-9	Coal tar-Baker fine	5	BCL	Heavy coating very, very heavy alligatoring healed, soft rubbery	Extremely alligator, pubby blisters
21, 22	CTC	Coal tar-Control	0	Commercial	Very smooth, high gloss, black medium soft	Bronzed flat, pinholed thin
23, 24	----	Coal tar-Baker fine	25	Phoenix	Slightly gray very rough, rough chunks	Very rough, no checks, no pinholes
27, 28	----	Coal tar-Union Carbide	5	Phoenix	Black, smooth hard, flat	Black, Smooth, few pinholes
29, 30	AC	Asphalt-Union Carbide	25	Phoenix	Medium cracking slightly gray soft, rubbery thin film	Considerable checking, gray, thin-eroded

(2) The Weather-O-Meter samples are applied to Transite® board (asbestos-cement composition) rather than blacktop.

(3) The Weather-O-Meter samples are subject to sudden temperature changes of wider excursion than the driveway samples.

(4) The Weather-O-Meter samples are not subject to the pressure of traffic, which can cause a healing of small cracks to occur.

Even though the Weather-O-Meter exposures were exhibiting atypical failures, they were continued on exposure to obtain valuable comparative data.

After only 100 hours of exposure, it became apparent that cracking of coal-tar-rubber blends was related to the amount of rubber in the blend. Cracking was restricted to the coal tar samples containing only 5 percent rubber. Those samples containing 25 percent rubber did not crack. Although no cracking has been observed in any samples exposed in the Battelle driveway, the results indicate that a higher amount of rubber than 5 percent might be desirable in coal tar compositions, in order to obtain maximum flexibility.

The differences observed early in the exposure period continued throughout the exposure life.

Slip Resistance of Experimental Emulsions

The experiment emulsions were brushed onto a damp piece of 1/8-inch asbestos board. Three specimens were evaluated for each experimental emulsion and a control. The slip resistance was measured in wet and dry conditions. The test procedure was to apply a piece of tread from a rubber tire to the coated surface and to place a 500-gram weight on the rubber tread. A Chatillon measuring device was used to determine the pull in pounds necessary to move the weighted rubber tread from at rest to a sliding position. The rubber tread had an area of 1.68 square inches. The measuring device is calibrated in 0.05-lb increments and is Model No. DPP-5. Table VI lists the specimens and the pull in pounds necessary to break friction hold, wet and dry. The figure given represents an average of three readings per specimen and the average of the three specimens evaluated.

TABLE 6. SLIP RESISTANCE OF EXPERIMENTAL EMULSIONS

Specimen No.	Description	Average pull, lb Dry	Wet
TE-1	5% Xylos 1x-Asphalt	1.27	1.19
TE-2	5% Baker fine-Asphalt	1.49	1.40
TE-3	5% Union Carbide-Asphalt	1.83	1.27
TE-4	25 % Baker fine-Asphalt	Unable to evaluate because of large chunks of rubber on surface of coating	
TE-5	5% Baker fine-Asphalt	1.61	1.49
TE-6	5% Xylos 1x-Coal tar	1.07	0.96
TE-7	5% Union Carbide Coal tar	1.09	1.05
TE-8	25% Union Carbide Coal tar	1.05	0.99
TE-9	5% Baker fine Coal tar	1.04	1.04
TE-10	Asphalt Control	1.43	1.56
TE-11	Coal tar Control	0.99	0.91

All of the experimental coal tar-rubber emulsions have improved slip resistant both wet and dry when compared to the commercial control. The same is true with the experimental asphalt-rubber emulsions with one exception. The asphalt emulsions all show greater skid resistance than the coal tar emulsions. This effect is mostly likely due to the slightly softer films of asphalt.

Application of Nine Experimental and Two Control Emulsions

The nine experimental and two control emulsions were applied on an evaluation site in the north drive of Battelle's Columbus Laboratories' entrance to the parking area. (See Figure 1.)

Most of the experimental emulsions were applied with no difficulty. Emulsion TE-4, Baker fine rubber at 25 percent level, was fairly hard to apply in a smooth coating because the rubber particles had swollen and agglomerated during standing in the container. Emulsion TE-8 was very difficult to apply because of a very rapid break time. Several of the experimental emulsions were gray when dried, indicating that too much extender material (talc and clay) was used in the formulation.

The emulsions were applied on Saturday, May 20, 1972, between 1:00 p.m. and 4:00 p.m. The temperature was between 80 and 85 F during the time of application. Figure 2 shows the evaluation site after application of the experimental and control emulsions. It is estimated that a minimum of 1800 cars pass over the experimental emulsions every weekday.

Three-Week Evaluation of the Applied Experimental Emulsions

The experimental emulsions and the two controls were given brief observations three weeks after their application. It is estimated that approximately 28,000 cars had passed over the experimental emulsions in this time period. Table 7 lists the emulsions and observations made.

TABLE 7. THREE-WEEK EVALUATION OF EXPERIMENT AND CONTROL EMULSIONS

Emulsion	Observations
TE-1 Asphalt 5% Xylos 1x	Very good crack sealing Not very soft Slightly streaky application
TE-2 Asphalt 5%	Grayish-white at edges Black in traffic areas Thick spots very soft Streaky application

FIGURE 1. EVALUATION SITE BEFORE APPLICATION

FIGURE 2. EVALUATION SITE AFTER APPLICATION

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TABLE 7. (Continued)

Emulsion	Observations
TE-3 Asphalt 5% Union Carbide	Tire abrasion marks Gray Thick areas soft Fair crack sealing Slightly streaked from application
TE-4 Asphalt 25% Baker	Grayish-white Very granular, rubbery, sticky Very streaky, Rubber appears to be separating
TE-5 Asphlat 5% Baker	Gray Tire abrasion marks Fairly tough Uniform appearancy Very slightly granular
TE-6 Coal tar 5% Xylos 1x	Very dark gray Thick areas slightly soft Cracks not very well sealed oil oil drippage resulted in soft spots
TE-7 Coal tar 5% Union Carbide	Black Very good general appearance Thick areas soft Cracks appear well sealed
TE_8 Coal tar 25% Union Carbide	Considerable tire abrasion marks Harder than TE-7 Considerably grayer than control Oil drippings not soft White in nontravelled areas
TE-9 Coal tar	Very thick spots are soft Few tire abrasion marks Dark gray-darker than asphalt control
Asphalt Control	Puddled Soft in deep areas Tire abrasion marks Slight gray general appearance
Coal tar Control	Very uniform Fair cracking filling Fairly hard in thick area

The observations made on the control and experimental emulsion after three weeks were very general and the differences were slight. No major trends were detected at that time.

Subsequent Evaluations

Periodic observations of the conditions of the applied stripes were made throughout the period from May 20, 1972, through May 20, 1973.

On July 28, 1972, observations indicated that all samples were showing wear on the tops of aggregate particles, including both controls. All samples continued to exhibit good sealing between aggregate particles. No major differences were noted between samples. Those samples appearing white or light gray shortly after application appeared to be darkening, so that very little difference (if any) could be seen between exposed samples.

Examination again in late December, 1972, showed some differences beginning to appear. However, they were decided to be not definitive enough to permit identification of any trends. Those samples containing 25 percent rubber, based on the bitumen content, appeared to be losing some of the granules of rubber by its working out of the surface, particularly in the high-traffic areas.

It also appeared at this time that the finer ground (cryogenic) rubber produces a more penetrating dressing that appeared to seal cracks better than those dressings made from coarser rubber particles.

Examination of the test patches was again made on March 20, 1973. In general, the winter of 1972-73 had been moderate, with only a small amount of snowfall in Columbus, Ohio. These conditions did, however, result in an unusual amount of freezing and thawing. In spite of this, all coatings including the controls appeared to have lasted through the winger remarkably well. Although the tops of the aggregate particles were worn clean of the dressing material, all coated areas, regardless of the coating material, were still well sealed between the aggregate particles, providing water-tightness. The conclusion was reached that all experimental samples were at least as good as the controls. Indications continued to be present that the 5 percent rubber was giving superior performance to the 25 percent rubber samples, and that the finer-ground rubber had apparently penetrated further into the pores and cracks of the substrate.

A final examination was made on May 22, 1973, after the applications had been on exposure for one full year. During that year, a conservative estimate indicated more than 450,000 vehicles had passed over the center portions of the applications. Although most of these vehicles were passenger cars, a significant number were trucks, many of them of the heavy, tractor-trailer type. The area also included a portion of each application exposed to parking, including oil drippage (on the south edge).

Prior to examination, the strips were hosed with cold water, and brushed dry with a push-broom, to insure the removal of loose dirt. The strips were also photographed when dry. These photographs are presented as Figures A-1 to A-11 in the Appendix of this report.

Observation of the general appearance of the exposed samples showed that all materials were in fair conditions. Although the sealer had been worn off the tops of aggregate particles, the area between particles remained filled, and the surface appeared to be well sealed for all samples. Cracks in the pavements that were present at the time of application either remained filled and sealed, or had not enlarged from their conditions prior to sealing.

Intercomparing the samples, all experimental materials appeared to be in at least as good condition as either of the two controls. The asphalt control appeared slightly more worn than the coal tar control.

Sample TE-1 remained quite smooth. The large crack in the base pavement remained fairly well sealed by the sealer. It had not enlarged from the pre-application state. It was not injured in the parking area in the foreground of the oil drippage.

Sample TE-2 showed some application marks due to thick areas. However, it remained in very good condition, and appeared to still be giving good protection and sealing.

Sample TE-3 appeared very uniform in application. It remained dark. No damage could be seen due to oil drippage.

Sample TE-4 showed marked application marks due to thick areas. The granular appearance that was evident just after application has, for the most part, disappeared. Due to its heavy nature, it did not penetrate the large crack very deeply. However, the crack still appeared sealed for the most part, and had not enlarged noticeably from its original size.

Sample TE-5 still retained its original uniform appearance. It remained dark, having lost most of the original gray appearance. Good protection of the base asphalt seemed to be maintained.

Sample TE-6 shows some application marks. The large crack is incompletely sealed, probably because of its large size. Generally good protection is being maintained by the coating.

Sample TE-7 is, for the most part, quite uniform. A few application marks can be seen in the foreground. Small cracks appear to be well sealed. The light spots in the foreground are due to oil drippage from parked cars. No injury to the coating was noted in these areas.

Sample TE-8 has lost its early whiteness and has now become dark gray. Very few application marks can be seen. Excellent protection seems to be maintained. Small cracks remain well sealed.

Sample TE-9 remains gray in color. Some application marks are visible. The large crack in the foreground remains well sealed. Generally good performance was noted.

Sample CTC, the coal tar control, appears very thin. The large crack at the left of the application was still well sealed. Show-through of the aggregate was very noticeable.

Sample AC, the asphalt control, appears slightly worn in the center, high traffic area. The large crack on the right remained well sealed.

CONCLUSIONS

Many conclusions are possible from the study as described in this report. However, it appears that the following are among the most important.

(1) It has been demonstrated beyond doubt that it is feasible to incorporate ground rubber from discarded automobile tires in bitumens such as coal tar or asphalt to prepare a product suitable for use as a blacktop dressing.

(2) The economics, although not completely resolved, seem to be favorable since it appears possible to improve the performance of asphalt to essentially match that of coal tar.

(3) The economics will, of course, depend on the optimum level of rubber in bitumen.

(4) The optimum level of rubber appears to lie somewhere between 5 and 25 percent of the rubber-bitumen blend.

(5) The rubber may be incorporated into the bitumen by several processes. Both the Battelle and the Phoenix process used in this study appeared to give satisfactory results.

(6) Satisfactory durability was observed for all experimental samples exposed after one year of service.

(7) Assuming 10 percent rubber to be the optimum concentration, each gallon of emulsion would contain approximately 1/2 pound of ground rubber. This gallon would normally cover about 40 square feet (optimistic estimate).

One passenger car tire, after removal of the bead and the major part of the fabric, will yield approximately 10 pounds of ground rubber, or enough to prepare 20 gallons of emulsion.

The average driveway is estimated to contain about 800 square feet (8' x 100'), and should require about 20 gallons of dressing for good protection.

It, therefore, appears that about one tire per average driveway would be used.

In addition, the millions of square feet of blacktop in shopping center parking lots, city streets, airport loading aprons, taxi strips, etc, could furnish enough area to use up all the roughly 200 million discarded passenger car tires per year.

PATENTS

It is believed that the process for emulsification of a vulcanized tire rubber-bitumen blend is new and novel. Before proposing the research program covered by the subject grant, a fairly comprehensive literature study failed

to uncover any reference to such an emulsification. The early phases of the study included contacts with many asphalt, coal tar, and rubber specialists. These contacts failed to reveal any emulsification of rubber-bitumen blends.

Although no patent novelty search has been made of the process, the intelligence obtained from the above-noted sources substantiates that the emulsification of rubber-bitumen blends is a novel process, resulting in a new, useful material that might be patentable.

After examining the many facets of this situation, the following course of action is recommended.

(1) The first step should be a patent novelty search of the process of preparing vulcanized rubber-bitumen (asphalt or coal tar) blends, and emulsification of such blends to prepare water-thinnable systems. This step would permit a decision regarding the possible patentability of the process, and the advisability of preparing an application for a patent.

(2) The preparation of a strong patent application will doubtless require some additional studies to establish limits of composition and preferred formulas. It should be remembered that the study covered by this report was designed to demonstrate feasibility of the basic idea. Neither time nor funds were available to obtain the comprehensive information and mutitude of examples, required for a strong patent application. Since the present contract has terminated, and no extension is being anticipated, funds for supporting these additional studies will need to be sought elsewhere.

(3) Assuming a patent application is made and a patent is granted for the process or product, the rights thereto would be governed by the patent provisions in the grant.

Since the fundamental purpose of this study is to stimulate widespread interest in and use of this method of solving one difficult phase of the urgent solid waste disposal problem, it is urged that any obtained patent become an item of public domain, in order to encourage as widespread use of the process as possible.

A report of invention has been prepared and is being submitted to the Grants Officer. This invention report covers the basic concept of blending ground automobile tires with bitumens (asphalt or coal tar) and emulsifying the resulting blends to form water-thinnable compositions suitable for use in treating blacktop driveways, parking lots, highways, etc.

FUTURE WORK

An inspection of the exposure series was made with the Project Officer of this study on May 24, 1973. At that time, it was mutually agreed between the Project Officer and Battelle's Columbus Laboratories' personnel that the objective of the present study had been reached. Complete feasibility of the basic idea of bitumen-rubber blends that could be emulsified had been demonstrated. Therefore, the objectives of the grant were judged to have been met, and no further work was judged to be necessary.

However, the complete success of this study has opened up many intriguing possibilities.

First and foremost, it must be emphasized strongly that the materials exposed in this study represent only starting point recipes. The limited program did not permit (1) selection of the optimum raw materials used to make the emulsions, (2) establishment of optimum rubber-bitumen ratios, (3) identification of best preparation techniques, or (4) conduct long-range storage stability tests. All of these studies should be carried out before suitable assurance of a marketable product can result.

In addition, the observed properties of rubber-bitumen blends point towards many possibilities for other uses than blacktop dressings. These might include

(1) Vehicle under-coats
(2) Coatings for underground protection of steel structures (tanks, pipes, etc.)
(3) Roofing (composition and built-up)
(4) Marine paints
(5) Adhesives
(6) Acid-proofing (battery cases, etc.)
(7) Waterproofing

(8) Expansion joints (concrete construction)

(9) Sound and vibration dampeners

(10 Sealants

(11) Caulking compounds

(12) Paper impregnants

(13) Ammunition case enamels

(14) Thermal insulation

Each of the above end-uses would require study to develop optimum compositions.

Considerable interest in this program has been exhibited by both government agencies and industrial organizations. The successful completion of a study such as this can be expected to result in aiding the solution of a solid waste disposal problem, and at the same time, development of a saleable product of improved quality and favorable economics.

APPENDIX

A-1

FORMULA TE-1

Water	9 kilograms
Mistron ZCX talc	3 kilograms
Calcium Chloride	60 grams
Asphalt - 5% rubber mix	4.5 kilograms

Asphalt - 4.275 kg
Xylos 1x - 0.225 kg

FORMULA TE-2

Water	9 kilograms
Mistron ZCS talc	3 kilograms
Calcium Chloride	60 grams
Asphalt - 5% rubber mix	4.5 kilograms

Asphalt - 4.275 kg
Baker Fine - 0.225 kg

FORMULA TE-3

Water	9 kilograms
Mistron ZCS talc	3 kilograms
Calcium Chloride	60 grams
Asphalt - 5% rubber mix	4.5 kilograms

Asphalt - 4.275 kg
Union Carbide - 0.225 kg

A-2

FORMULA TE-4

Water	9 kilograms
Mistron ZCS talc	3 kilograms
Calcium Chloride	60 grams
Asphalt - 25% rubber mix	4.5 kilograms

Asphalt - 3.375 kg
Baker Fine - 1.125 kg

FORMULA TE-5

Water	7.2 kilograms
Burgess #20 Clay	1.2 kilograms
Mistron ZCS talc	0.6 kilograms
Indulin W-1	60 grams
Calcium Chloride	60 grams
Potassium ricinoleate	60 grams
Coal tar - 5% rubber mix	5.4 kilograms

Coal tar - 5.130 kg
Baker Fine - 0.270 kg

FORMULA TE-6

Water	7.2 kilograms
Burgess #20 Clay	1.2 kilograms
Mistron ZCS talc	.6 kilograms
Indulin W-1	60 grams
Calcium Chloride	60 grams
Potassium ricinoleate	60 grams

FORMULA TE-6 (Continued)

Coal tar - 5% rubber mix	5.4 kilograms
Coal tar - 5.130 kg Xylos 1x - 0.270 kg	

FCRMULA TE-7

Water	7.2 kilograms
Burgess #20 Clay	1.2 kilograms
Mistron ZCS talc	0.6 kilograms
Indulin W-1	60 grams
Calcium Chloride	60 grams
Potassium ricinoleate	60 grams
Coal tar - 5% rubber mix	5.4 kilograms
Coal tar - 5.130 kg Union Carbide - 0.270 kg	

FORMULA TE-8

Water	7.2 kilograms
Burgess #20 Clay	1.2 kilograms
Mistron ZCS talc	0.6 kilograms
Indulin W-1	60 grams
Calcium Chloride	60 grams
Potassium ricinoleate	60 grams
Coal tar - 25% rubber mix	5.4 kilograms
Coal tar - 4.05 kg Union Carbide - 1.35 kg	

A-4

FORMULA TE-9

Water	7.2 kilograms
Burgess #20 Clay	1.2 kilograms
Mistron ZCS talc	0.6 kilograms
Indulin W-1	60 grams
Calcium Chloride	60 grams
Potassium ricinoleate	60 grams
Coal tar - 5% rubber mix	5.4 kilograms

Coal tar - 5.130 kg
Baker Fine - 0.270 kg

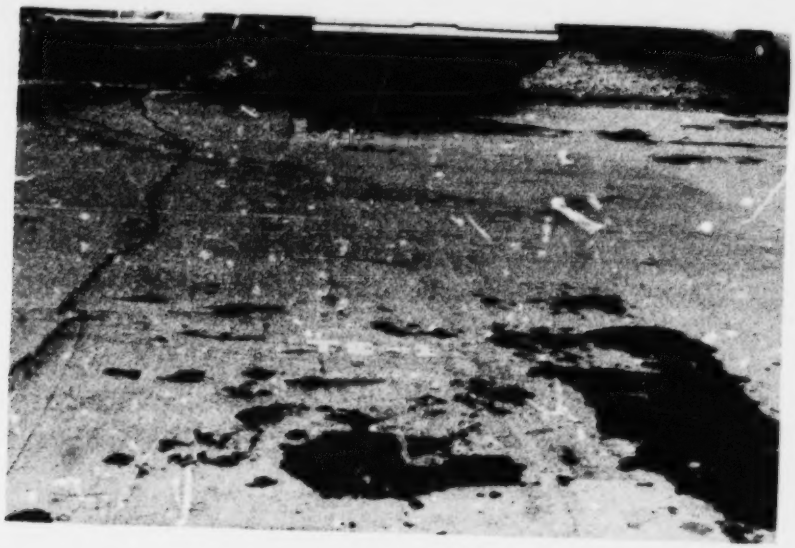

FIGURE A-1. 1 YEAR EXPOSURE - SAMPLE TE-1

FIGURE A-2. 1 YEAR EXPOSURE - SAMPLE TE-2

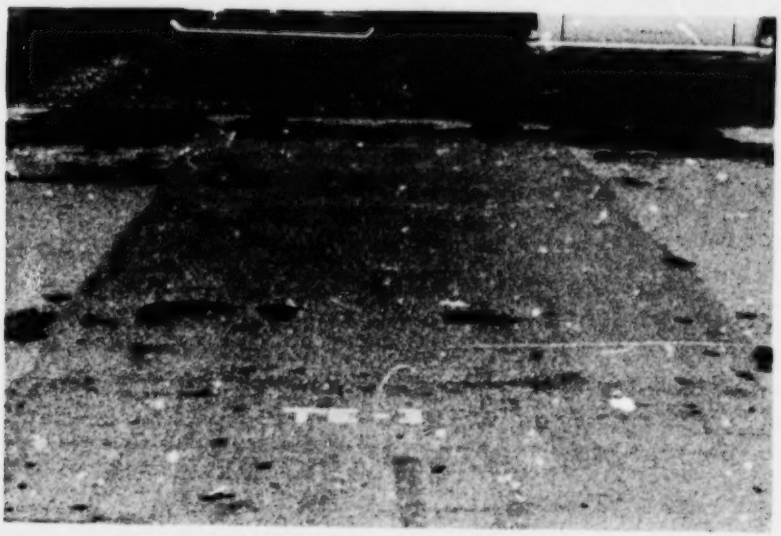

FIGURE A-3. 1 YEAR EXPOSURE - SAMPLE TE-3

FIGURE A-4. 1 YEAR EXPOSURE - SAMPLE TE-4

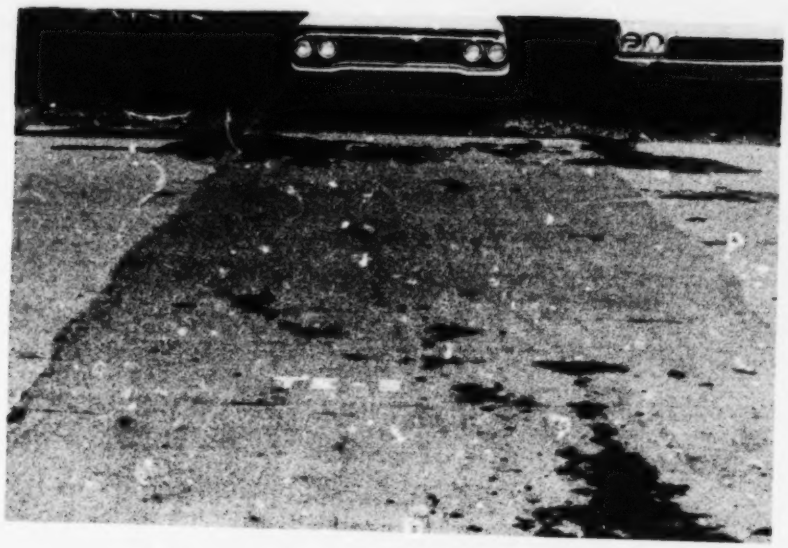

FIGURE A-5. 1 YEAR EXPOSURE - SAMPLE TE-5

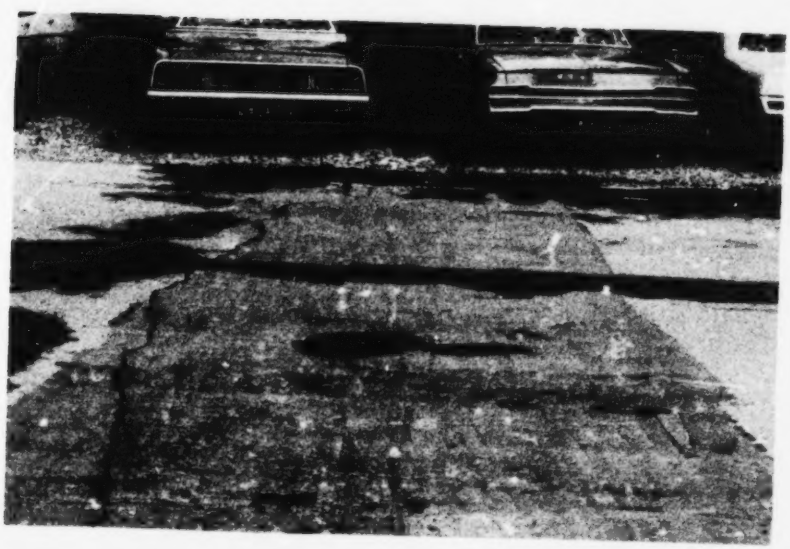

FIGURE A-6. 1 YEAR EXPOSURE - SAMPLE TE-6

FIGURE A-7. 1 YEAR EXPOSURE - SAMPLE TE-7

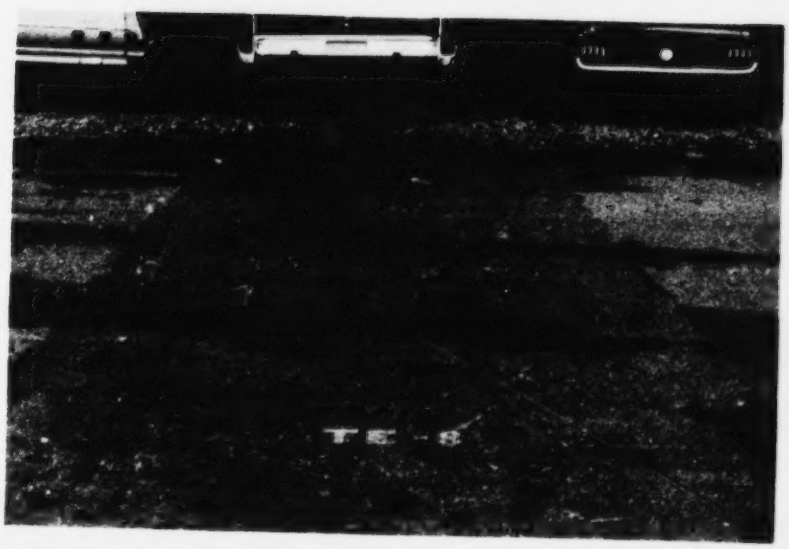

FIGURE A-8. 1 YEAR EXPOSURE - SAMPLE TE-8

FIGURE A-9. 1 YEAR EXPOSURE - SAMPLE TE-9

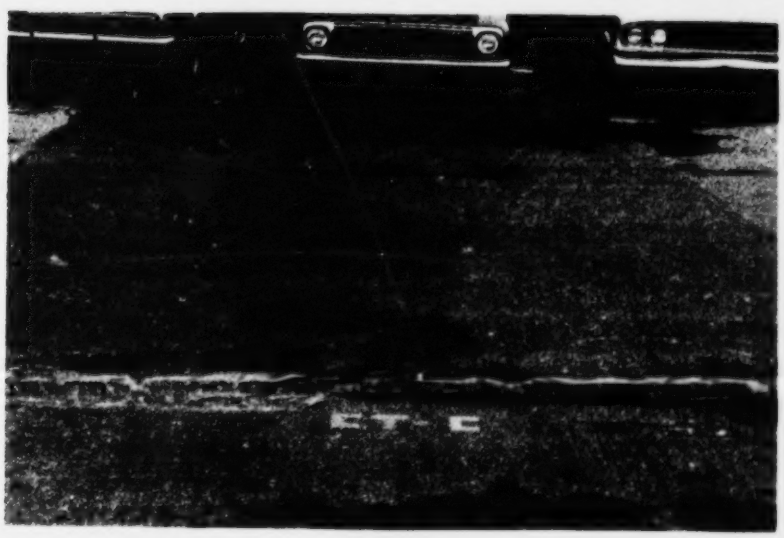

FIGURE A-10. 1 YEAR EXPOSURE - SAMPLE CT-C

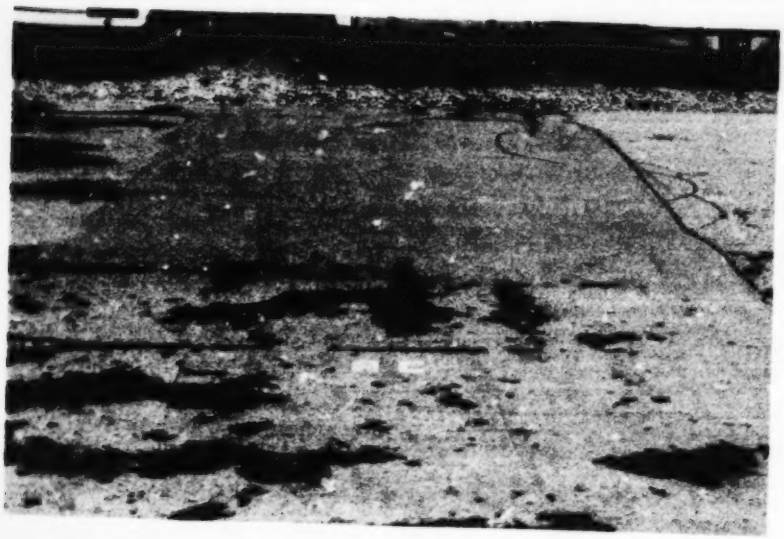

FIGURE A-11. 1 YEAR EXPOSURE - SAMPLE AC